

NASA TECHNICAL NOTE



NASA TN D-6794

e. f.

NASA TN D-6794

LOAN COPY: RETURN
AFWL (DOUL)
KIRTLAND AFB, N. M.



METHOD FOR MEASURING STATIC YOUNG'S MODULUS OF TUNGSTEN TO 1900 K

by William T. Harrigill, Jr., and Alois Krsek, Jr.

Lewis Research Center

Cleveland, Ohio 44135



0133739

1. Report No. NASA TN D-6794		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle METHOD FOR MEASURING STATIC YOUNG'S MODULUS OF TUNGSTEN TO 1900 K				5. Report Date April 1972	
				6. Performing Organization Code	
7. Author(s) William T. Harrigill, Jr.; and Alois Krsek, Jr.				8. Performing Organization Report No. E-6702	
				10. Work Unit No. 114-03	
9. Performing Organization Name and Address Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135				11. Contract or Grant No.	
				13. Type of Report and Period Covered Technical Note	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D. C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes					
16. Abstract An instrument system was developed and tested to measure static Young's modulus of elasticity of refractory metals in tension at temperatures to 1900 K. The extensometer uses capacitance displacement sensors for measuring total elongation of a specimen and incorporates a unique high-resolution remote zero-adjusting mechanism. Details of a method for calibrating capacitance displacement sensors at temperatures to 800 K are included. The system was built to adapt to a vacuum furnace on a tensile machine and was tested on unalloyed tungsten rod specimens. The test results show that the system can measure the modulus of tungsten to 1900 K to within a precision of ± 5 percent. The uncertainty of the measurement is estimated to be of the same order as the precision.					
17. Key Words (Suggested by Author(s)) Temperature; High temperature; Elongation; Metals; Tungsten; Strain; Stress; Refractory metals; Extensometer; Tensile modulus; Young's modulus; Capacitance proximity meter				18. Distribution Statement Unclassified - unlimited	
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of Pages 16	
				22. Price* \$3.00	

METHOD FOR MEASURING STATIC YOUNG'S MODULUS OF TUNGSTEN TO 1900 K

by William T. Harrigill, Jr.; and Alois Krsek, Jr.

Lewis Research Center

SUMMARY

An instrument system was developed and tested to measure static Young's modulus of elasticity of refractory metals in tension at temperatures to 1900 K. The extensometer uses capacitance displacement sensors for measuring total elongation of a specimen and incorporates a unique high-resolution remote zero-adjusting mechanism. Details of a method for calibrating capacitance displacement sensors at temperatures to 800 K are included. The system was built to adapt to a vacuum furnace on a tensile machine and was tested on unalloyed tungsten rod specimens. The test results show that the system can measure the modulus of tungsten to 1900 K to within a precision of ± 5 percent. The uncertainty of the measurement is estimated to be of the same order as the precision.

INTRODUCTION

Advanced nuclear-fueled space power systems rely heavily on the use of refractory metals and their alloys because of the very high temperatures encountered in these systems. A necessary part of the materials work in support of the space power program is the determination of the properties of applicable alloys over the expected system temperature range. An especially difficult problem is the measurement of Young's modulus of elasticity because strain measurements are required at temperatures far in excess of the capabilities of conventional strain-measuring instrumentation. The purpose of this work was to develop an instrument system to measure the static Young's modulus of refractory metals at temperatures to 1900 K.

References 1 and 2 describe two methods that have been used to measure Young's modulus at high temperature. The more conventional technique (ref. 1) involves measuring the elongation of a specimen under tensile stress by using a remote optical tracking device. The method of reference 2 (called the dynamic method) involves calculating Young's modulus from the velocity of propagation of a stress wave through the material.

The velocity of propagation is measured by determining the resonant frequency of a simple vibrational mode.

The instrument system reported herein follows the more conventional technique of reference 1 and was built to adapt to an existing tensile test machine. This machine had previously been modified for tensile testing of refractory alloys at high temperature. The test specimens were swaged tungsten rods. Specimen elongation was measured with an extensometer developed in this program which uses capacitance proximity sensors.

The following sections contain a discussion of the choice of measuring technique; a description of the measuring system and the methods used to calibrate the proximity sensors; and a discussion of the results of tests on the system, including data on Young's modulus of unalloyed tungsten rod to 1900 K.

MEASUREMENT TECHNIQUE

The conventional method for measuring modulus was chosen for this work because this method tests a specimen under conditions which approximate the conditions under which the material will be used (i. e., in static structures). The choice of measurement method is important because existing modulus data produced by the static and dynamic methods do not agree at high temperature. For example, reference 1 gives the modulus of tungsten sheet at 1900 K at 190 GN/m^2 . The "dynamic" modulus for tungsten rod at 1900 K from reference 3 is given as 325 GN/m^2 , roughly 1.7 times the "static" modulus.

The reason for this difference in results is not clear. The following factors have been suggested (refs. 1 to 3) as possibilities:

(1) The vibrational frequencies involved in the dynamic method are high, of the order of 10^4 hertz. This implies that adiabatic conditions exist in the specimen. A conversion to isothermal conditions has been suggested (ref. 2) but the corrections for tungsten at 1900 K are insufficient to resolve the difference.

(2) The specimens tested were different: the "static" modulus measurement was made on commercially prepared sheet, and the dynamic measurement was made on swaged rod. Variations in impurity level and differences in structure and/or anisotropy induced by specimen-forming techniques may contribute to this difference.

(3) In the static method, specimen creep may obscure the measurement of elastic strain. This effect would tend to produce low modulus data, in agreement with the direction of the differences noted. However, in the work of reference 1, stress level was limited (14 MN/m^2 at 1900 K) and test times kept short in order to preclude specimen creep.

In the instrument system reported herein, the tensile load and the specimen elongation are plotted on a recorder. The slope of this load-against-elongation line, modified by appropriate scale factors for the length and area of the specimen, gives the modulus.

An extensometer was developed to measure the specimen elongation. This extensometer transfers the relative motion of the ends of the specimen out of the high-temperature region to a cooler area, where it can be measured. This transfer is accomplished with tungsten rods welded to each end of the test specimen. The rods extend out of the heated area into a cooler region, where their relative displacement is measured by a capacitance-type proximity meter (refs. 4 and 5). A capacitance-type proximity meter was selected for this application because it incorporates a noncontacting displacement sensor capable of operating accurately at elevated temperatures.

SYSTEM DESCRIPTION

A sketch of the test chamber of the tensile testing machine (ref. 6) is shown in figure 1. As shown in the sketch a test specimen of the material is attached to the force column by means of a pair of tungsten pull rods and split-grip assemblies. The specimen is surrounded by a heater, a heat shield assembly, and a water-cooled jacket. All these components are enclosed in a water-cooled vacuum chamber. The force train is constructed with a steel ball-and-socket arrangement to allow for self-alignment of the force column and to generate only tensile forces as load is applied to the specimen. The crosshead (to which the force column is tied) is driven at constant speed by means of a servocontrol system incorporated in the tensile machine. The ambient temperature outside the heat shield stabilizes between 600 and 650 K when the specimen is heated to 1900 K.

The test specimens (fig. 2) were short rods fabricated from swaged unalloyed tungsten (made by the powder metallurgy process), 39 millimeters in active length and 4 millimeters in diameter. The radius of curvature of the test specimen between the active length and the clamping heads was made small in order to clearly define the active length of the test specimen.

Figure 3 is a schematic drawing of the extensometer. As shown in the figure tungsten rods are welded to the top and bottom of the specimen. These tungsten (extension) rods are extended through the center of hollow tungsten pull rods out beyond the heated area into the cooler section of the vacuum chamber. The upper extension rod is attached to a steel frame which transfers the motion of the top rod down to close proximity with the end of the lower extension rod. Any change in the length of the specimen is thus indicated as a differential movement between the end of the lower extension rod and the lower frame bar. Because of the heat conducted down the lower extension rod, the tip of the rod and the adjacent region develop temperatures of more than 800 K for a specimen

temperature of 1900 K. Therefore, the relative movement of the extension rods is transferred laterally by the rocker arm mechanism (1:1 ratio arms) to a still lower-temperature region. The rocker arm is counterweighted to ensure good contact between the rocker pin and the lower extension rod. This movement, which is indicative of the total elongation of the test specimen, is measured as a gap change between the specimen capacitance sensor and its target plate. The counterweight mounted to the lower frame bar is used to establish the extensometer's center of gravity along the longitudinal axis of the specimen.

The fine zero-adjusting mechanism (also shown in fig. 3) remotely changes the specimen sensor-to-target-plate gap with a resolution of the order of 0.025 micrometer ($1\text{ }\mu\text{in.}$) during operation in a vacuum at elevated temperatures. This mechanism consists of two stainless-steel tubes and an additional capacitance-type proximity meter. When the metal tubes are electrically heated, their thermal expansion changes the position of the mounting block to which the specimen target plate is mounted. The target plate is moved toward or away from the specimen sensor depending on which of the tubes is heated. By placing the zero-adjusting proximity meter in the feedback loop of an operational amplifier (fig. 4), the position of the specimen target plate relative to the lower frame bar can be remotely positioned and automatically controlled. The metal tubes act as resistance heaters. Any difference between the output of the zero-adjusting proximity meter and the set point voltage is amplified and, depending upon the polarity of this signal, causes a change in current to the appropriate heater tube. This change in current positions the target plate to the desired position. The total adjustment is approximately ± 100 micrometers.

The following coarse zero-adjustment technique is used to correct for large (about 2 mm for a specimen temperature of 1900 K) changes in the sensor-to-target-plate gap size. An approximate magnitude of the change is calculated from the thermal coefficient of expansion of the materials and the temperature of the particular experiment to be run. A spacer equal in thickness to the calculated gap change is placed between the rocker pin and the tip of the lower extension rod while the specimen sensor is manually adjusted to within its operating range at room temperature. The spacer is then removed. As a result, the specimen sensor rests on the target plate. When the specimen is heated to its predetermined temperature, the rocker pin will be forced downward, causing the rocker arm to rotate counterclockwise and restore the gap to approximately its room-temperature value. Any further adjustment is accomplished by the fine zero-adjusting mechanism. Figure 5 shows the test chamber and the extensometer.

PROXIMITY SENSOR CALIBRATION

In order to provide extensometer accuracy at all temperatures that might be encountered during testing, the sensors were calibrated at room temperature, 500 K, and 800 K.

For the room-temperature calibration a system was used which consisted of a Michelson interferometer (ref. 7) and a means of generating linear mechanical displacements. These displacements are accurately measured with the interferometer by observation of light fringes. A mercury light source and filter combination provide a light fringe every 0.2730 micrometer. The reticle of the telescope used to observe the light fringes provides a means of estimating fractional parts of a light fringe. The specimen sensor was calibrated at intervals of 10 light fringes (2.730 μm) over a range of 130 micrometers (0.005 in.), producing a calibration with a sensitivity of $1\frac{1}{2}$ millivolts per micrometer and a linearity of about $\pm 3\frac{1}{2}$ percent to within a precision (1 sigma) of ± 0.076 micrometer (± 3 $\mu\text{in.}$).

At elevated temperatures the specimen sensor was checked by means of a device shown in figure 6 which generates linear motion through the thermal expansion of stainless-steel tubes (heater tubes). Voltage is applied across these tubes which act as resistance heaters. Thermal expansion forces the upper mounting plate upward away from the common mounting plate, generating a gap displacement for the upper and lower capacitance sensors (decreasing the size of the upper gap while increasing the size of the lower gap). The upper sensor (previously calibrated with a Michelson interferometer) is used to accurately measure this displacement from which the lower sensor can be checked. The upper sensor remains at room temperature while the lower sensor is heated. The heat supplied to the heater tubes did not appreciably affect the length of the lower target support tube during a calibration run.

The effect of room-temperature change on the upper gap is minimized by fabricating the heater tubes and the upper target support tube from the same type of material (stainless steel) and cutting them to approximately the same length. In this way the same thermal expansion (or contraction) occurs in all three tubes, keeping the sensor gap approximately constant. The same principle applies to the lower sensor gap. By using the upper sensor as a secondary standard to measure displacement, a calibration of the lower sensor was made at room temperature, 500 K, and 800 K over a 25-micrometer range. Five runs (made over this range at each temperature) were averaged. The results showed that the greatest change occurred at 800 K. The scatter in data increased from ± 0.3 percent at room temperature to ± 0.6 percent, and the sensitivity increased by 0.4 percent.

SYSTEM TESTING AND RESULTS

Preliminary Room-Temperature Tests

The system was tested initially at room temperature by means of a torque arm load-cell calibrator. The force train used to apply load to the tungsten specimen was the same type used for high-temperature testing. A force of 2700 newtons (used as full-scale loading in all room-temperature tests) produced a stress of about 200 MN/m^2 , causing the specimen to elongate approximately 20 micrometers. A load cell located in series with the force train measured the specimen loading to within ± 10 newtons. The output of the load cell, along with the output of the extensometer, was recorded as a continuous plot of force against elongation. Young's modulus was then determined from the slope of this curve.

A total of over 100 tests were conducted on the load-cell calibrator using three different tungsten specimens at room temperature to give an average modulus of 392 GN/m^2 .

From these tests the precision of a single observation (1 sigma) was determined to be ± 1 percent. The room-temperature modulus value was verified by measuring strain as indicated by the average reading of two strain gages diametrically mounted on the specimen. The average reading of the two strain gages was used in order to minimize the effect of any slight bending of the specimen. The uncertainty in the strain gage measurements was about $\pm 3/4$ percent. The average modulus as determined by the strain gages was 397 GN/m^2 (approximately $1\frac{1}{4}$ percent higher than that measured by the extensometer).

During room-temperature testing it was observed that a misalignment of the force train caused a nonlinearity in the stress-strain curve in the vicinity of zero loading. When this occurred, a small tare weight (200 N or less) corrected this condition.

High-Temperature Tests

A second series of tests were conducted in much the same manner as the initial testing except they were conducted in the tensile testing machine (previously described) at temperatures to 1900 K.

In these tests the load was applied linearly to the specimen starting from zero (or a small tare weight), increased to a predetermined maximum value, and then immediately reduced in the same manner to its original value. A continuous plot of force against elongation was recorded during the load-unload procedure.

Three tungsten specimens were involved in these tests, although most of the data were obtained from only two of the specimens. The first specimen was used primarily to develop the required experimental techniques involved in high-temperature experiments. This included determining the maximum allowable stresses to be applied while testing the

specimens at elevated temperatures. It was necessary to reduce the maximum applied stress as the temperature was increased to prevent a permanent set due apparently to a combination of creep and yield strength characteristics of the material. The maximum stress used in this testing at 1900 K was 17 MN/m^2 , which limited the maximum elongation to about 2.5 micrometers ($100 \mu\text{in.}$) or approximately one-eighth of the elongation at room temperature. During the load-unload cycle the tensile machine held the strain rate approximately constant. Rate of specimen elongation varied between 0.1 and 1 micrometer per second. For all tests above 800 K, 1 micrometer per second was used. A minimum warmup period of 4 hours was allowed prior to a series of tests (taken at a particular temperature) to ensure thermal stabilization.

The results of these tests are given in table I. The average modulus, the number of readings, and the precision (1 sigma) of a single observation are listed for each temperature at which the specimens were checked. The uncertainty of the measurements is

TABLE I. - MODULUS OF ELASTICITY MEASUREMENTS
OF UNALLOYED TUNGSTEN

Temperature, K	Average modulus of elasticity, \bar{X}		Number of readings, n	Standard deviation ^a , percent
	GN/m ²	psi		
300	395	57.4×10^6	18	± 1.0
810	378	54.8	49	± 4.3
1090	349	50.6	8	± 2.8
1360	322	46.8	28	± 3.4
1640	276	40.1	23	± 4.0
1900	224	32.6	9	± 5.4

$$^a \text{Standard deviation} = \sqrt{\sum_i \frac{(X_i - \bar{X})^2}{n - 1}} / \bar{X}, \text{ where } X_i \text{ is modulus}$$

reading, \bar{X} is average modulus, and n is number of readings.

estimated to be of the same order as the precision. These results are shown in figure 7 as a plot of Young's modulus against temperature. Both the average values and the maximum spread in readings are given in this plot. Figure 7 also shows data obtained by the dynamic method (ref. 3) and the outside boundaries (shaded area) of measurements taken on thin sheet specimens by at least 10 different experimentors (refs. 1 and 8). It can be seen that the data of the present work falls in between that of reference 3 and that of references 1 and 8. At 1900 K, the data of this report are only 10 percent higher than the upper extreme of the results of reference 8 (static method), although they are 50 percent higher than the mean of references 1 and 8. The results of figure 7 should be taken only

as a general comparison because of the variance between the present work and references 1, 3, and 8 involved in generating the results. These were (1) different methods of testing (dynamic and static), (2) difference in specimen geometry (rod and sheet), and (3) variation in material structure due to different fabrication techniques.

SYSTEM LIMITATIONS

There were limitations in the system used in this work which should be avoided because they contribute to the scatter in the data. These limitations were as follows:

(1) There was a mismatch between the tensile machine and the loading required by the tungsten specimen. The machine had a maximum load capacity of 45 kilonewtons, while the load requirement at 1900 K was about 220 newtons.

(2) The coarse zero adjustment was awkward and time consuming.

(3) A slight mechanical coupling between the high-temperature sensor cable and the vacuum chamber walls caused a slight nonlinearity in the specimen's elongation-load curve.

SUMMARY OF RESULTS

A system incorporating a capacitance-type extensometer in a high-temperature tensile testing machine was successfully used to measure the modulus of elasticity of tungsten to 1900 K. The technique used in this work is feasible for determining Young's modulus to 1900 K to within a precision (1 sigma) of ± 5 percent. The accuracy is estimated to be of the same order as the precision. Modulus values for tungsten were found to be 395 GN/m^2 at room temperature and 224 GN/m^2 at 1900 K. These static values are higher than previous static determinations but fall in between results obtained from the dynamic method and these previous static measurements.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, February 18, 1972,
114-03.

REFERENCES

1. Zocher, R. W. : High Temperature Measurement of Young's Moduli for Tungsten and Tungsten-Uranium Dioxide Composites. Union Carbide Corp. (NASA CR-54847), Dec. 9, 1965.
2. Neudecker, J. W. ; and Saunders, C. R. : Young's Modulus Measurements to 2500^o C by the Thin-Rod Resonance Technique. Advances in Instrumentation. Vol. 24, pt. 2. Inst. Soc. Am., 1969, pp. 582.1-582.4.
3. Armstrong, Philip E. ; and Brown, Harry L. : Dynamic Young's Modulus Measurement Above 1000^o C on Some Pure Polycrystalline Metals and Commercial Graphites. Trans. AIME, vol. 230, no. 5, Aug. 1964, pp. 962-966.
4. Hartman, D. E. ; Bresie, D. A. ; and Roberts, J. M. : Capacitance Extensometers for Microstrain Measurements. Rev. Sci. Instr., vol. 34, no. 11, Nov. 1963, pp. 1272-1273.
5. Anon. : A Capacitance-Type Transducer for Absolute Measurement of Displacements. DISA Information, July 1965, pp. 20-24.
6. Raffo, Peter L. ; Klopp, William D. ; and Witzke, Walter R. : Mechanical Properties of Arc-Melted and Electron-Beam-Melted Tungsten-Base Alloys. NASA TN D-2561, 1965.
7. Strong, John: Concepts of Classical Optics. W. H. Freeman and Co. , 1958, p. 381.
8. Schmidt, F. F. ; and Ogden, H. R. : The Engineering Properties of Tungsten and Tungsten Alloys. DMIC Rep. 191, Battelle Memorial Institute, Sept. 27, 1963. (Available from DDC as AD-425547.)

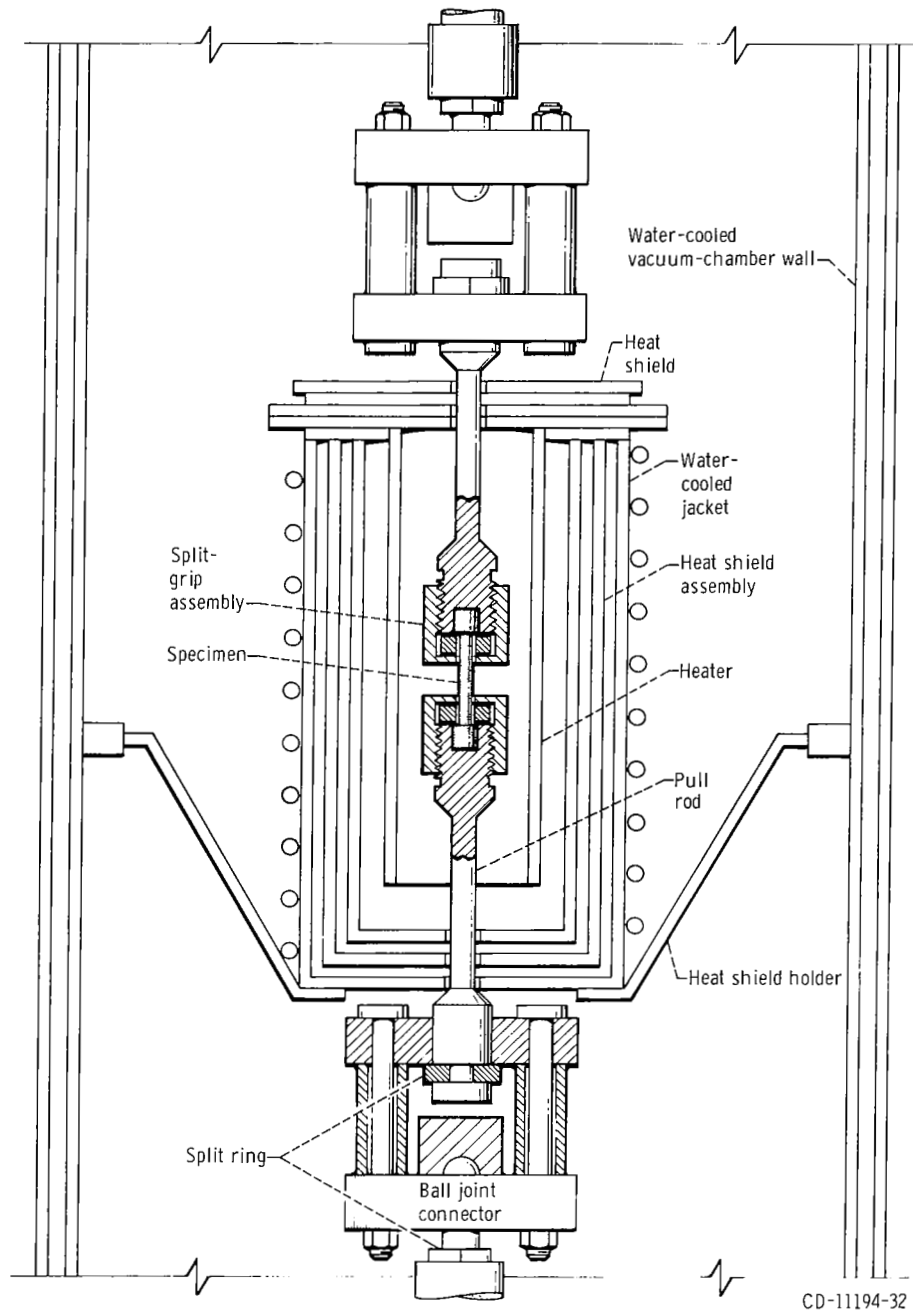


Figure 1. - Tensile machine test chamber.

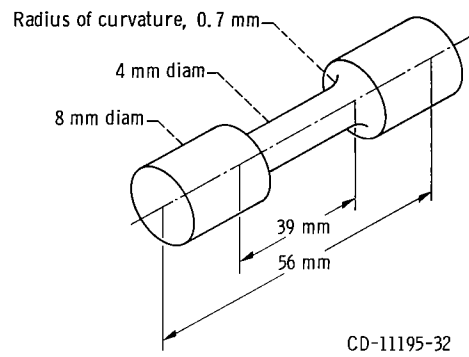
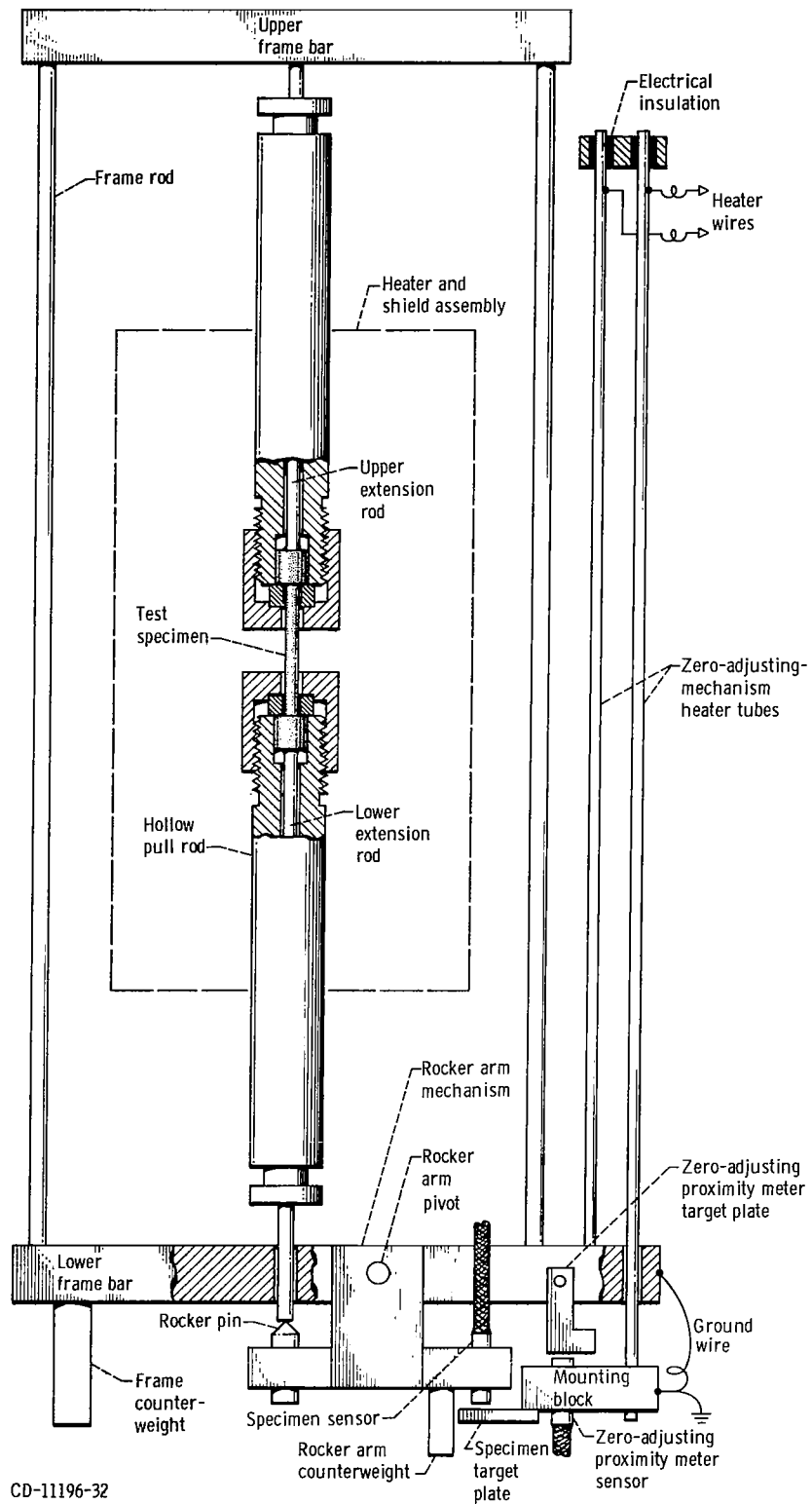


Figure 2. - Tungsten test specimen.



CD-11196-32

Figure 3. - Extensometer.

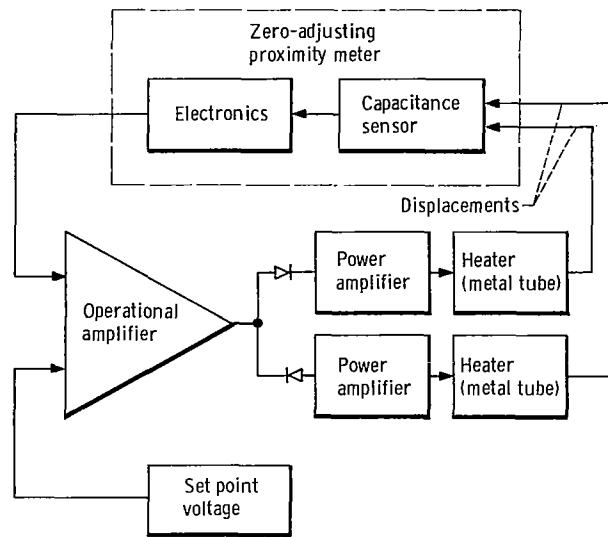
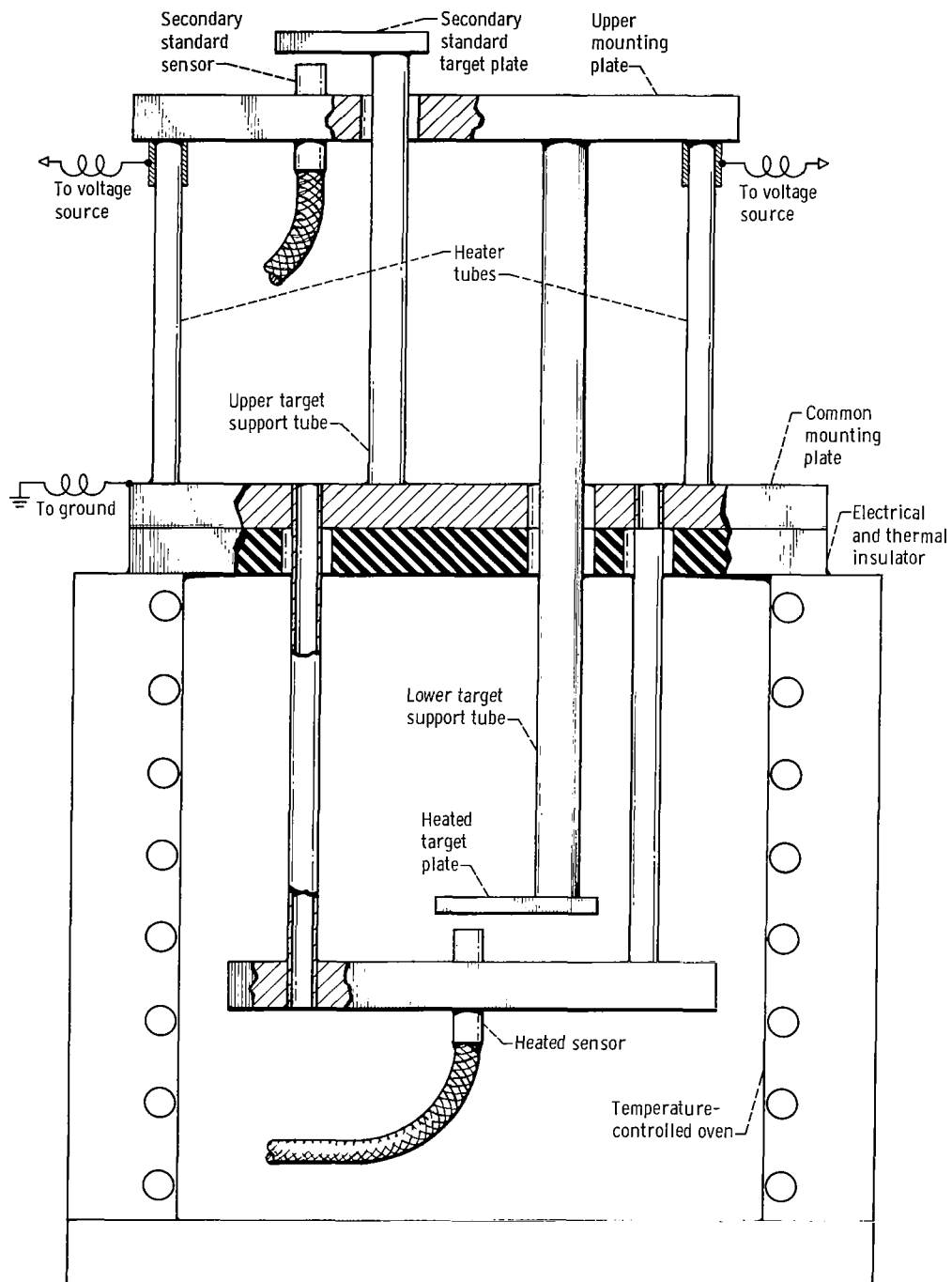


Figure 4. - Zero-adjusting control circuit.



Figure 5. - Extensometer and test chamber.



CD-11197-32

Figure 6. - High-temperature displacement calibrator.

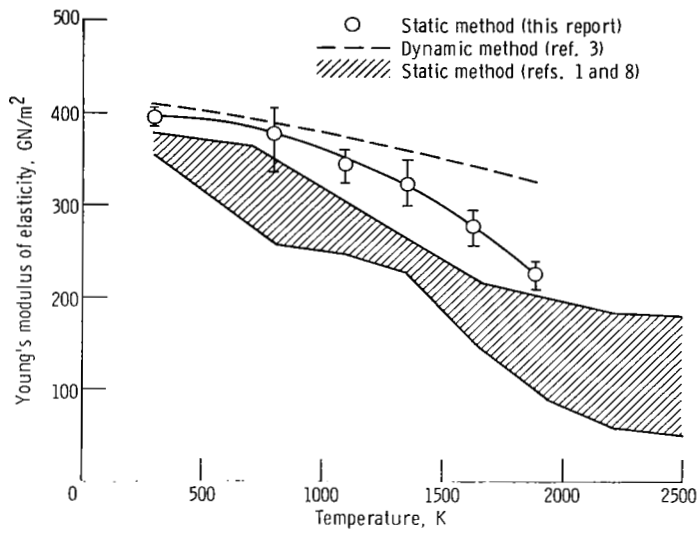


Figure 7. - Young's modulus of elasticity as function of temperature for unalloyed tungsten.